SLOVENSKI STANDARD
SIST EN 12441-2:2004

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Cink in cinkove zlitine ï Kemiļ na analiza ï 2. del: Doloļ evanje magnezija v cinkovih zlitinah ï Metoda plamenske atomske absorpcijske spektrometrije

Zinc and zinc alloys - Chemical analysis - Part 2: Determination of magnesium in zinc alloys - Flame atomic absorption spectrometric method

Zink und Zinklegierungen - Chemische Analyse - Teil 2: Bestimmung von Magnesium in Zinklegierungen - FAAS-Verfahren

Zinc et alliages de zinc - Analyse chimique - Partie 2: Dosage du magnésium dans les alliages de zinc - Méthode par spectrométrie d'absorption atomique dans la flamme

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ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.120.60	Svinec, cink, kositer in njihove zlitine	Lead, zinc, tin and their alloys

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Zinc and zinc alloys - Chemical analysis - Part 2: Determination of magnesium in zinc alloys - Flame atomic absorption spectrometric method

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This European Standard was approved by CEN on 11 August 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 209, "Zinc and zinc alloys", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

Within its programme of work, Technical Committee CEN/TC 209 entrusted CEN/TC 209/WG 6 “Methods of analysis and testing”, with the preparation of the following document:

prEN 12441-2, *Zinc and zinc alloys - Chemical analysis - Part 2: Determination of magnesium in zinc alloys – Flame atomic absorption spectrometric method.*

This standard is part of a series of eleven standards. The other standards are:

— *Part 1 : Determination of aluminium in zinc alloys - Titrimetric method*

— *Part 3 : Determination of lead, cadmium and copper - Flame atomic absorption spectrometric method*

— *Part 4 : Determination of iron in zinc alloys - Spectrophotometric method*

— *Part 5 : Determination of iron in primary zinc - Spectrophotometric method*

— *Part 6 : Determination of aluminium and iron - Flame atomic absorption spectrometric method*

— *Part 7 : Determination of tin - Flame atomic absorption spectrometric method after extraction*

— *Part 8 : Determination of tin in secondary zinc - Flame atomic absorption spectrometric method*

— *Part 9 : Determination of nickel in zinc alloys - Flame atomic absorption spectrometric method*

— *Part 10 : Determination of chromium and titanium in zinc alloys – Spectrophotometric method*

— *Part 11 : Determination of silicon in zinc alloys - Spectrophotometric method*

The annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a flame atomic absorption spectrometric method for the determination of magnesium in zinc alloys. It is applicable to the products specified in EN 1774 and EN 12844.

It is suitable for the determination of magnesium contents (mass fractions) between 0,002 % and 0,08 %.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1774, *Zinc and zinc alloys - Alloys for foundry purposes - Ingot and liquid.*

EN 12060, *Zinc and zinc alloys - Method of sampling – Specifications.*

EN 12844, *Zinc and zinc alloys - Castings - Specifications.*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions.*

3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in EN 12060 and the following term and definition apply.

3.1
flame atomic absorption spectrometry
measurement of the absorption of electromagnetic radiation, emitted by an element at a determined wavelength, by an absorbent medium (flame) formed of atoms of the same element that are in the ground state. Each element absorbs radiation of specific wavelengths and the intensity of the absorbed radiation is proportional to the concentration of said element

4 Principle

A sample of the alloy is dissolved in a mixture of hydrochloric and nitric acid and, after adequate dilution and atomization of the solution in an air-acetylene (or nitrous oxide-acetylene) flame, the content of magnesium is determined by atomic absorption spectrometry at the wavelength of 285,21 nm.

5 Reagents

5.1 General

During the test, use only reagents of known or analytical grade and distilled or demineralised water.

5.2 Hydrochloric acid, ρ = 1,19 g/ml

5.3 Nitric acid, ρ = 1,4 g/ml

5.4 Hydrochloric acid - nitric acid mixture

Mix 180 volumes of hydrochloric acid (5.2) with 4 volumes of nitric acid (5.3). This mixture shall be freshly prepared just before use.

5.5 Lanthanum, 5 % solution

Introduce 29,5 g of lanthanum oxide (La_2O_3) in a 400 ml beaker. Add 5 ml of water, then carefully add 50 ml of hydrochloric acid (5.2). After dissolution, cool to room temperature. Transfer quantitatively to a 500 ml volumetric flask. Dilute to the mark with water and mix.

5.6 Zinc, 10 g/l solution

Dissolve 10 g of zinc (99,99 %), free from magnesium, (see 5.11), with 60 ml of the acid mixture (5.4). Evaporate to a syrupy consistency. Take up with water and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

5.7 Aluminium, 1,0 g/l solution

To 1,0 g of aluminium (99,99 %), free of magnesium, (see 5.11), add 10 ml of water and then dissolve with the minimum of hydrochloric acid (5.2). Heat gently to aid dissolution. Cool to room temperature. Transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

5.8 Magnesium, 0,5 g/l solution

Into a 250 ml beaker covered with a watch-glass, pour 20 ml of water, then 5 ml of the hydrochloric acid (5.2). Introduce 0,5 g of magnesium of purity at least 99,95 %, weighed to ± 0,001 g. After dissolution of the metal, cool and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

5.9 Standard magnesium solution A

Transfer exactly 20 ml of the magnesium solution (5.8) to a 1 000 ml volumetric flask. Add 5 ml of the hydrochloric acid (5.2). Dilute to the mark with water and mix.

1 ml of this solution contains 0,01 mg of magnesium.

5.10 Standard magnesium solution B

Transfer exactly 50 ml of the standard magnesium solution A (5.9) to a 500 ml volumetric flask. Add 5 ml of the hydrochloric acid (5.2). Dilute to the mark with water and mix.

1 ml of this solution contains 0,001 mg of magnesium.

5.11 Aqua regia

Mix 3 volumes of hydrochloric acid (5.2) with 1 volume nitric acid (5.3).

6 Apparatus

6.1 General

All glassware used for the preparation of the solutions and for the implementation of the method shall be cleaned with boiling aqua regia (5.11) prior to use.

6.2 Specific equipment

In addition to standard laboratory apparatus, an atomic absorption spectrometer, equipped with a premix burner, with facilities for using the oxidizer–fuel combinations of air–acetylene or nitrous oxide–acetylene, shall be used.

NOTE Excitation sources should be operated in accordance with the manufacturer's recommendations. The optical path length within the flame should be between 5 cm and 10 cm.

7 Sampling

The test sample shall be selected and prepared in accordance with the procedure given in EN 12060.

8 Procedure

8.1 Test portion

Weigh 5 g of the test sample to the nearest 0,001 g.

8.2 Preparation of the test solution

8.2.1 Introduce the test portion (8.1) into a 250 ml beaker fitted with a watch-glass and dissolve by carefully adding 40 ml of the acid mixture (5.4). Evaporate carefully to a syrupy consistency and, after cooling to room temperature, take up with 40 ml to 50 ml of water. Add 25 ml of the hydrochloric acid (5.2) and warm gently to dissolve any salts.

8.2.2 Transfer to a 250 ml volumetric flask. Dilute to the mark with water and mix.

8.2.3 Transfer exactly 10 ml of this solution (8.2.2) to a 100 ml volumetric flask. Add 4 ml of the hydrochloric acid (5.2) and 5 ml of the lanthanum solution (5.5). Dilute to the mark with water and mix.

8.3 Preparation of the calibration solutions

8.3.1 To verify that the magnesium content of solution (5.6) or (5.7) is low enough, proceed as follows:

— introduce into two 100 ml volumetric flasks 0 ml and 2 ml respectively of the standard magnesium solution B (5.10) corresponding to 0 mg/l and 0,02 mg/l of magnesium;

— dilute to the mark with water and mix;

— compare solution (5.6) or (5.7) with these calibration solutions by spectrophotometric measurement of the atomic absorption as specified in 8.4.

The spectrometric measurement response shall not exceed that of the 0,02 mg/l solution.

8.3.2 Introduce 5 ml of the hydrochloric acid (5.2) into each flask of a series of eight 100 ml volumetric flasks.

8.3.3 Add a ml of the zinc solution (5.6) and b ml of the aluminium solution (5.7) to each flask, according to Table 1.

Table 1 — Volumes *a* and *b*

Aluminium content (mass fraction) %	*a* ml	*b* ml
Smaller than 0,05	20	0
Between 3,7 and 6,0	19	10
Between 8,0 and 11,0	18	20
Between 25,0 and 28,0	15	50

8.3.4 Then add 0,00 ml, 2,00 ml, 5,00 ml, 7,00 ml, 10,0 ml, 12,0 ml, 14,0 ml and 16,0 ml aliquots of the standard magnesium solution A (5.9). These aliquots correspond to contents (mass fractions) in the test portion of 0,00 %, 0,01 %, 0,025 %, 0,035 %, 0,05 %, 0,06 %, 0,07 % and 0,08 % of magnesium.

For the analysis of products ZL6 and ZP0610, prepare 2 additional calibration solutions corresponding to magnesium contents (mass fractions) of 0,002% and 0,005 %, by taking respectively 2,00 ml and 5,00 ml aliquots of the standard magnesium solution B (5.10).

8.3.5 Add 5 ml of the lanthanum solution (5.5) to each flask. Dilute to the mark with water and mix.

8.4 Spectrometric measurements

Measure the absorbances of the calibration solutions and the test solution(s) by taking alternate readings to ensure that the settings of the burner and of the apparatus do not change during the readings.

The wavelength of the line used shall be 285,21 nm.

To comply with the concentration ranges recommended by the manufacturer of the apparatus, the same dilutions for the calibration solutions and the test solution(s) shall be made if necessary.

NOTE To obtain better reproducibility and greater sensitivity, it is recommended that a slightly reducing flame be used.

9 Calculation and expression of results

9.1 Method of calculation

Determine from the measured absorbance of the test solution the associated amount of magnesium from the calibration graph. If a number of determinations are carried out then the mean of all results shall be calculated by adding the individual results together and dividing by the number of individual results.

The results shall be expressed as specified in EN 1774 and EN 12844.

9.2 Precision

Repeatability and reproducibility limits are given in Table 2.

NOTE Table 2 has been established according to annex A. Graphical representation of precision data are given in annex B.